

CHARACTERISTICS OF A HERMETIC 6H-SiC PRESSURE SENSOR AT 600°C

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ABSTRACT

We report the fabrication and characterization of a single crystal hermetically sealed 6H-SiC pressure sensor tested up to 600°C. The pressure sensor was packaged by a novel microelectromechanical systems direct chip attach (MEMS-DCA) technique that eliminated the use of wire bonding, thereby removing the reliability issues associated with wire bonds at high temperature. The room temperature full-scale output (FSO) at 200 psi was 32.5 mV for a bridge input voltage of 5V, which gives a sensitivity of 32.5 μ V/V/psi. Although the full-scale output at 600°C dropped by 64 %, it maintained excellent linearity, with less than 2 mV null shift upon return to room temperature.

INTRODUCTION

There is growing demand for improved fuel efficiency in jet engines and automobiles as well as the reduction of undesirable emission of hydrocarbons and other combustion by-products such as NO_x and CO. Furthermore, as modern engine design increases its reliance on computer modeling, the validation of the computer codes in actual engine testing is recognized to be crucial. Without such validation, these codes may not

be trustworthy for use in future engine designs.

The temperature of the instrumentation of combustion engine environments is typically greater than 300°C. However, it is challenging to apply conventional semiconductor electronic and sensing devices because they are limited to operating temperatures less than 300°C due to the limitations imposed by their material properties and traditional packaging technology. To circumvent the thermal barrier, complex and costly packaging has been implemented, which includes plumbing of the package to allow cooling fluid to remove the heat. This allows devices based on silicon-on-insulator (SOI) technology to be used in extending the thermal ceiling to about 400°C,

Silicon carbide has long been viewed as a potentially useful semiconductor for high temperature applications. Its excellent electrical characteristics - wide-bandgap, high-breakdown electric field, and low leakage current- make it a superior candidate for high-temperature electronic applications¹. Furthermore, SiC exhibits excellent thermal and mechanical properties at high temperature and fairly large piezoresistive coefficients, a combination that makes it well-suited for high temperature electromechanical sensors^{2,3}. SiC-based electronics and sensors have been demonstrated to operate at temperatures up to 600°C⁴, thereby offering promise of direct

insertion into the high temperature environment without cooling. However, the lack of reliable device packaging methodologies for this operating environment has largely prevented the application of these devices. As a result, the anticipated introduction of SiC devices into high temperature environments has been delayed.

In recognition of the package limitation, research efforts have intensified within the harsh environment sensors and electronic community to develop robust packaging that will support the reliable operation of these devices. Several methods to achieve robust packaging for high temperature pressure sensors have been proposed^{5,6}. While most of these are at different stages of development, initial feasibility has been demonstrated in an engine⁷.

Although SiC electronic and sensing devices targeted for use in environments of greater than 300°C possess the desired properties, the functionality must satisfy the necessary reliability criteria before acceptance. Devices capable of functioning in these harsh environments need the appropriate package to sustain operation throughout its entire planned life cycle. Therefore, in the absence of robust and reliable packaging, these devices are useless if they cannot perform and endure in the designated harsh environment. An effort at NASA Glenn Research Center has adopted a packaging strategy that de-couples the thermochemical and thermomechanical interactions between the sensor, metallization, and packaging components. As a result, the behavioral characteristics of the sensor itself at high temperature can be better understood without much influence from other components.

In this paper, we will present preliminary results of this strategy that has led to the development of a new generation of single crystal SiC MEMS based pressure transducers that we believe will be capable of long-term operation at 600°C in air.

SENSOR FABRICATION

High resistivity p-type 3.5° off-axis (0001) 6H-SiC wafers with 2-μm thick n-type 6H-SiC epilayers were purchased from Cree, Inc.⁸. The doping level of the epilayer was greater than $2 \times 10^{19} \text{ cm}^{-3}$. After backside polishing and visual inspection, the wafer was cleaned successively in acetone and methanol to remove organic particulates from the surface. To pattern the piezoresistors, a nickel lift-off process was performed on the n-type epilayer with photoresist. After lift-off, the nickel left on the wafer acted as an etch mask to define the pattern of the piezoresistors and the contact area. The wafer was reactively ion etched in a mixture of argon and NF₃ to etch off the n-type material not protected by the nickel mask, producing a patterned mesa of n-type SiC. The nickel was stripped from the wafer in a mixture of HNO₃, HCl, and H₂O (1:1:3) and rinsed in de-ionized water (DI-water).

The backside of the wafer (p-type) was coated with 6 μm thick photoresist and then circular patterns were defined and developed. Indium tin oxide (ITO) 3 μm thick was deposited onto the developed regions of the wafer. This was followed by the dissolution of the photoresist pattern, leaving exposed circles of SiC surrounded by ITO. Using the ITO pattern, a deep reactive ion etching process was then performed that etched an array of 250 μm deep circular cavities in the SiC as shown in Fig. 1a. The ITO that survived the etching process was stripped in H₂SO₄ and H₂O₂ solution (Piranha clean) and the wafer rinsed in DI-water. The wafer was then thermally dry-oxidized at 1150°C for 4 hours, after which the oxide was stripped in 49% HF acid, rinsed in DI water, and blow-dried with nitrogen. This step typically removes foreign

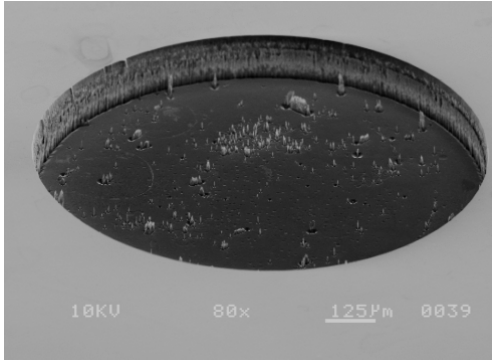


Fig. 1a: Scanning Electron Microscopy (SEM) micrograph of cavity etched in 6H-SiC by deep reactive ion etching method. Features on the surface are artifacts of ITO micromasking.

elements including chlorine that reside on the surface and a few nanometers into the epilayer, thereby leaving a cleaner surface.⁹ The samples then underwent another dry thermal oxidation at 1150°C for five hours to grow oxide about 70 nanometers thick on the wafer epilayer side.

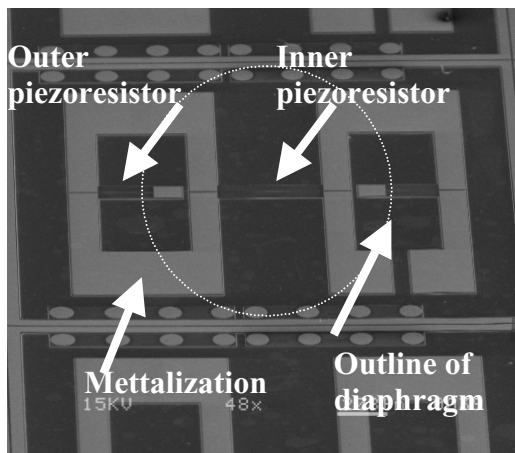


Fig. 1b: SEM micrograph of top view of a 6H-SiC pressure sensor cell with patterned metallization and four piezoresistors. The circular patterns are ohmic contact test structures.

Standard photolithography and oxide etching with buffered HF was used to etch contact vias in the oxide to expose the SiC only in the sections where high temperature ohmic metallization would be deposited.

After the oxide etching, the wafer was subsequently treated in another round of Pirhana-clean after which it was rinsed in DI water, blow-dried with nitrogen, and then transferred into the metallization chamber. A one-hour heat treatment at 300°C was performed in ultra high vacuum condition and cooled prior to depositing the multilayer metallization. After metal deposition, and etching of the contacts¹⁰, the wafer was annealed in nitrogen for 30 minutes at 600°C to make the contact ohmic. The piezoresistive mesas as shown in Fig. 1b were configured in a Wheatstone bridge circuit.

PACKAGE DESCRIPTION

In previous work¹¹, little consideration was given to package related thermally-induced stress in terms of its impact on the mechanical and electrical functionality and long-term reliability of the sensor. The existence of stress during thermal cycling is generally recognized to induce fatigue at several critical areas of the system, such as at the wirebond/pad interface. The packaging methodology adopted here borrowed from the traditional flip-chip bump packaging technology that allows a chip (or a chip array) to be intimately attached to another level of metallization via either a through-hole in package substrate or directly on corresponding interconnects on the package substrate. The approach eliminates wirebonds, thereby making it possible to pack chips more densely than previously possible.

The main objective of this work was to develop and demonstrate alternative methods of fabricating packages that provide environmental protection to electronics and sensors targeted for use in high temperature

environments. In principle, the common denominator of the schemes currently under investigation is that the sensor is thermomechanically de-coupled from the main stainless steel package to minimize

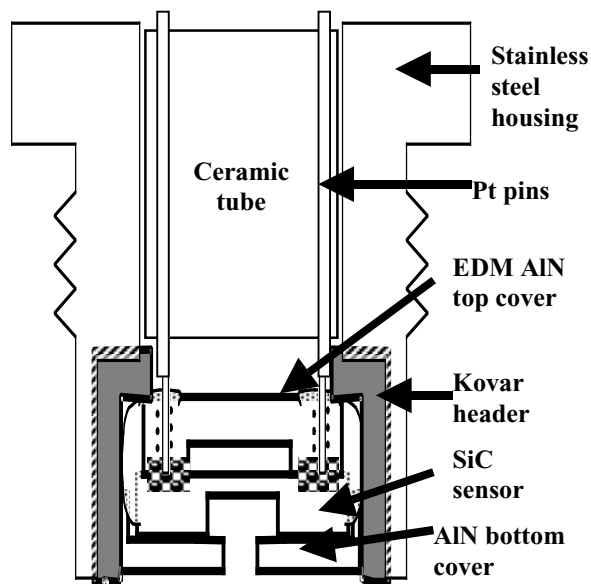


Fig. 2a: Sub-assembled 6H-SiC sensor unit prepared for insertion into the stainless steel screw housing.

undesired effects caused by large differences in the coefficient of thermal expansion between stainless steel and sensor. In the approach adopted here, the basic components of the package are shown in Fig. 2a, which consisted of a bottom substrate of an insulating dielectric material with thermomechanical properties similar or closely similar to that of silicon carbide (SiC) (i.e. aluminum nitride). This bottom cover substrate member served as a receiving platform for the SiC sensors. It also served as the first level of protection of the sensor from harmful particulates in the high temperature environment. The combination of the bottom cover together with the top cover substrate, made from the same material as the bottom substrate, served to provide sandwiched protection for the sensor. Because the covers were made of the same material with

thermomechanical properties similar to that of the sensors, the problem of mismatch in the coefficient of thermal expansion (CTE) associated with multi-component systems was significantly reduced. The top cover substrate has three important features. It has four through holes spread equidistantly. These holes accept wires or pins that are used to make intimate contact with the contact pads on the sensor. A shallow circular or rectangular recess is located on one face of the top cover substrate to provide over-pressure protection or a reference cavity. When the top cover substrate is placed on the bottom cover substrate, the recessed cavity in the top cover substrate faces down so that the

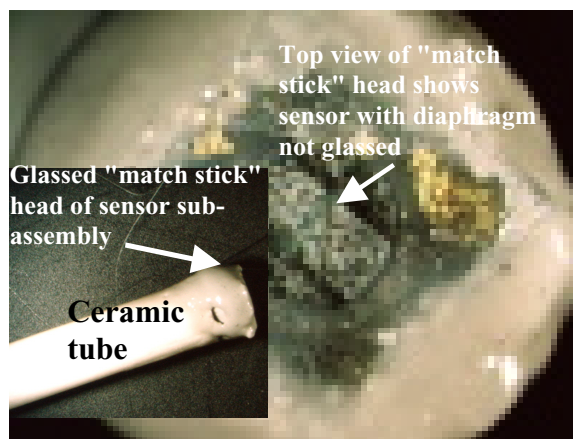


Fig. 2b: "Match-stick" type assembly in which the sensor sub-assembly is inserted into ceramic tube (inset) and then glass-encapsulated except for the diaphragm prior to insertion into stainless steel housing. This provided stress de-coupling between sensor and housing.

moving part of the sensor (i.e. diaphragm) resides within its peripheral boundary. High temperature glass was then applied over the sandwich unit to semi-encapsulate it, leaving only the diaphragm section as shown in Fig. 2b exposed to the environment. This process traps an air pocket within the reference cavity and provides hermetic sealing for the sensor after encasement between the top and bottom

cover substrates. The pins that are inserted into the through holes of the top cover substrate were made of platinum. This direct chip attach (DCA) process eliminates the need for wirebond and the associated failure mechanisms at high temperature.

Several approaches have been investigated for performing the final packaging of the sub-assemblies. In the approach applied here, the pins were inserted into a four-hole alumina ceramic tube. The section of the ceramic tube that rests against the back of the top cover piece was glassed and cured. This creates the resemblance of a match stick as shown in Fig. 2b. The sub-assembled unit was then inserted into the kovar header as illustrated in Fig. 2a. Prior to insertion, sealing glass was applied to the inner surface of the kovar header for

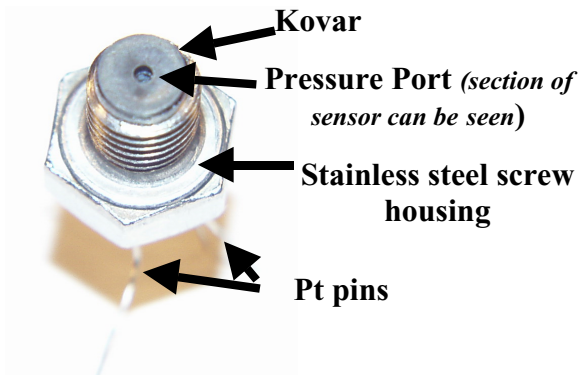


Fig. 2c: Fully packaged 6H-SiC pressure transducer with pressure port and pins visible.

temporary adhesion. The glass will eventually detach from the Kovar upon cooling. The stainless steel/kovar unit and the sub-assembled units are then fired at the glass cure temperature at 750°C for 15 minutes, which enables the bonding of the sensor unit and the Kovar header. A representative fully packaged transducer is shown in Fig. 2c

SENSOR CHARACTERISTICS

Generally, for a circular, edge fixed diaphragm (whose deflection is less than its thickness), the radial stress on the surface at any point is directly related to the applied pressure as expressed below¹²:

$$\sigma_r = \frac{3Pa^2}{4t^2} \left[(3m+1) \frac{r^2}{a^2} - (m+1) \right] + \sigma_p(T) \quad (1)$$

where σ_r = radial stress (psi), P = applied absolute pressure (psi), t = diaphragm thickness, a = diaphragm radius, r = distance from the center of a circular plate to the clamped edge, m = reciprocal of Poisson's ratio, ν . The extra term, $\sigma_p(T)$ on the right is author's insertion. It represents the coupled stress from the surrounding components. In a high temperature environment, σ_p will become dominant as a result of the differences in CTE of the components. Therefore, proper design consideration must be given to the choice of material and packaging complexity. In addition to closely matching the CTEs of the sealing glass (4.7 ppm/°C), 6H-SiC (4.2 ppm/°C) and AlN (4.1 ppm/°C)¹³, σ_p was greatly minimized during thermal cycling because the Kovar and stainless steel were de-coupled from the sensor sub-assembly.

For pressure applied on the cavity side of the diaphragm, as was the case here, the maximum tensile and compressive stress magnitudes occur at the diaphragm center and at its intersection with the edge, respectively. Therefore, in this design, the four piezoresistors were arranged longitudinally (radially) as shown in Fig. 1b. In that case, only a small transverse piezoresistance would be introduced while the longitudinal piezoresistance would dominate. The bridge output in terms of the radial stress and gauge factor can be expressed as:

$$\frac{\Delta V}{V} = \frac{\Delta R}{R} = \frac{\sigma_r}{E} GF \quad (2)$$

where E is the Young's modulus (psi) of 6H-SiC, which is approximately 65 Mpsi (448 GPa)¹². For the epilayer doping level used in this work, the gauge factor, GF , was estimated at 20, based on previous work⁴.

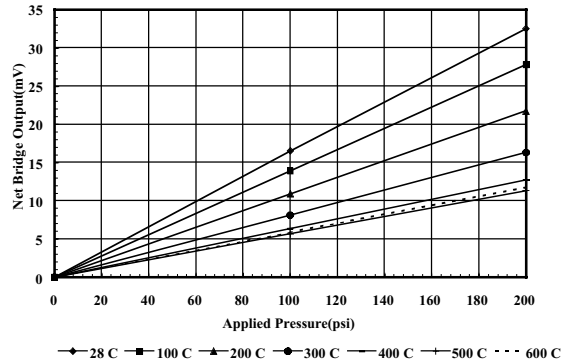


Fig. 3a: Net bridge output voltage of 6H-SiC pressure sensor as function of pressure at various temperature regimes.

Using equation (1), for the diaphragm radius of 600 μm and thickness of 50 μm , the predicted maximum stress magnitude was 21.95 kpsi (150 MPa), which is about 33 % of the fracture strength of SiC. Therefore, equation (2) predicts that the maximum output (assuming a balanced bridge) at 200 psi, for a 5 V excitation voltage will be 33.3 mV.

The net output voltage as a function of applied pressure at various temperatures is shown in Fig. 3a. The full-scale output (FSO) at a maximum applied absolute pressure of 200 psi was 32.5 mV at room temperature for an input voltage of 5V, which was in good agreement with prediction. This indicated a sensitivity of 32.5 $\mu\text{V/V/psi}$. The excellent linearity obtained could be largely attributed to the thermomechanical stress management adopted with the packaging, however, more data points will be needed to improve the measurement resolution. At 600°C, the full-scale output dropped to 11.76 mV, a 64 % decrease. The output of the transducer became less sensitive to temperature above 400°C, as

shown in Fig. 3b, which coincides with the temperature that the bridge resistance shown

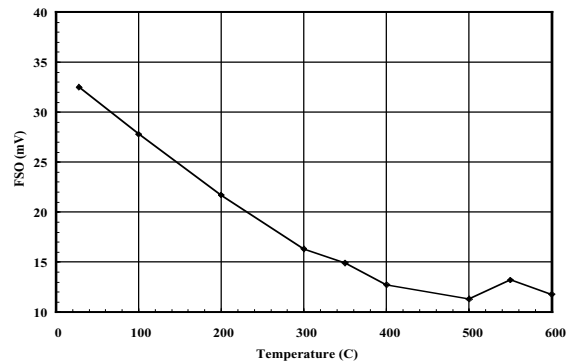


Fig. 3b: Full-scale output as function of temperature depicting the linear drop and eventual insensitivity to temperature.

in Fig. 3c began to increase. A high temperature compensation scheme is currently under development to address this problem. Upon cooling down, the bridge output at zero pressure shifted by 2 mV. While this shift is relatively small, its existence is consistent with the null shifts observed previously^{4, 11}. Although the source of the shift is not fully understood, component relaxation during thermal treatment could not be ruled out.

The effect of temperature on the resistance is shown in Fig. 3c. It indicates a gradual decrease from room temperature bridge output resistance of 145 Ω to about

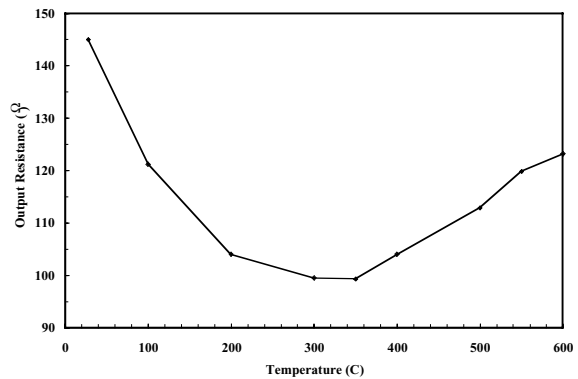


Fig. 3c: Bridge resistance of 6H-SiC piezoresistive pressure sensor as function of temperature ($N_d > 2 \times 10^{19} \text{ cm}^{-3}$).

99 Ω at 350°C due to increasing ionization of free carriers from SiC epilayer dopants atoms. The upward swing of the bridge resistance of the SiC is associated with the gradual dominance of lattice scattering mechanism over small further increase in carrier concentration with temperature¹⁴.

CONCLUSION

We have successfully demonstrated a hermetically sealed 6H-SiC pressure transducer that operated at 600°C and at 200 psi. This result demonstrates proof of concept of MEMS-DCA packaging methodology for high temperature applications. This methodology offers the following key unique features that enabled successful sensor operation at 600°C. The silicon carbide (SiC) semiconductor has similar coefficient of thermal expansion (CTE) as the AlN and glass. Because the sensor is sandwiched by AlN, very little CTE mismatch existed within the sub-system, thereby minimizing thermo mechanically induced stress transmitted to the sensor device at high temperature. The decoupling of the sub-system from the Kovar and stainless steel also enhanced stress reduction. In addition to the above, the metallization used was previously demonstrated to survive at 600°C in air for 1000 hours. While this packaging method is expected to enable extended high temperature device functionality, long-term reliability characterization will be performed in the future to validate such capability.

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